

Crystal structure of 4,4'-disulfanediyldipyridinium chloride triiodide

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Abstract

4,4'-Disulfanediyldipyridinium chloride triiodide (**1**) was synthesized by reaction of 4,4'-dipyridyldisulfide with ICl in 1:1 molar ratio in dichloromethane solution. The structural characterization of **1** by SC-XRD analysis was supported by elemental analysis, FT—IR, and FT-Raman spectroscopic measurements.

1. Chemical context

The reactions of pnictogen/chalcogen donors with dihalogens X_2 or interhalogens XY ($X, Y = \text{Cl, Br, I}$) afford a variety of products depending on the nature of the donor, the dihalogen/interhalogen, and the reaction conditions (Aragoni *et al.* 2008; Rimmer *et al.* 1998; Aragoni *et al.* 2022; Knight *et al.* 2012). For chalcogen donors, Charge-Transfer (CT) "spoke" adducts, hypercoordinate "T-shaped" adducts, halonium adducts, and different types of cationic oxidation products of the donors have been identified and structurally characterized (Knight *et al.* 2012; Saab *et al.* 2022). Worthy of note, diiodine CT-adducts have been extensively investigated, also with a view to their application as leaching agents for toxic (Isaia *et al.* 2011) and precious metals (Zupanc *et al.* 2022) in Waste from Electrical and Electronic Equipment (WEEE). Among the pnictogen donors, many studies have focused on (poly)pyridyl derivatives. Analogous to S/Se-donors, the reactions of pyridyl donors with X_2/XY resulted in the formation of CT-adducts featuring a linear $N\cdots X-Y$ group (Kukkonen *et al.* 2019; Tuikka & Haukka 2015) and halonium derivatives with an $N\cdots X^+\cdots N$ moiety ($X = \text{I}; Y = \text{Cl, Br, I}$) (Kukkonen *et al.* 2019; Batsanov *et al.* 2005; Batsanov *et al.* 2006). In addition, N-protonated pyridinium cations were obtained, whose charge can be counterbalanced by discrete halides or extended fascinating networks (Aragoni *et al.* 2004; Aragoni, Podda *et al.* 2023). Oxidation of the aromatic heterocycle to give a cationic radical species followed by solvolysis or reaction with incipient moisture has been proposed as a possible explanation for the formation of pyridinium cations (Rimmer *et al.* 1998; Aragoni, Podda *et al.* 2023).

The nature of the products isolated in the solid state is reflected in their peculiar FT-Raman response (Aragoni *et al.* 2008; Aragoni *et al.* 2004; Pandeewaran *et al.* 2009). In particular, an elongation of the perturbed $X-Y$ moiety with respect to the free halogen/interhalogen is found in CT-adducts, which determines a low energy shift of the relevant Raman-active stretching vibration (Aragoni *et al.* 2008). When polyhalide networks are formed, the stretching vibrations of the interacting synthons can be detected in the low-energy region of the FT-Raman spectrum (Aragoni *et al.* 2008; Aragoni, Podda *et al.* 2023).

Disulfides are an important class of organic compounds with a variety of biological and pharmacological applications (Sevier & Kaiser, 2002; Lee *et al.* 2013), in particular due to their antioxidant and prooxidant properties (Zhu *et al.* 2023). It is well known that the dibromine and dichlorine oxidation of diaryldisulfides leads to the cleavage of the sulfur–sulfur bond (Zincke reaction) (Zincke 1911; Baker *et al.* 1946), whereas the reaction of disulfides with the mildest oxidant diiodine, does not involve the cleavage of the S–S bond (Aragoni, Podda *et al.* 2023). The reaction of 2,2'-di-

40 pyridyldisulfide (**L**) with I₂ in CH₂Cl₂ afforded the compound [(HL⁺)(I⁻)·5/2I₂]_∞, featuring an unusual polyiodide network
41 counterbalancing the N-monoprotonated HL⁺ cation. Recently, an assembly isostructural to [(HL⁺)(I⁻)·5/2I₂]_∞ was ob-
42 tained by reacting 2,2'-dipyridyldiselenide with I₂ in either CH₂Cl₂ or CH₃CN (Aragoni, Podda *et al.* 2023).

43 Although 4,4'-dipyridyldisulfide (**L'**) has been widely reported as a donor towards a variety of metal ions (Sarkar *et al.*
44 2016; Zheng *et al.* 2022; Zheng *et al.* 2023; Singha *et al.* 2018), its reactivity towards halogens or interhalogens has been
45 only marginally explored (Wzgarda-Raj *et al.* 2021; Coe *et al.* 1997). An example is provided by
46 4,4'-(disulfanediyldipyridinium pentaiodide triiodide (CSD code OXAFIF) where the cation H₂L'²⁺ is counterbalanced
47 by a polyiodide built up of interacting I₃⁻ and I₅⁻ ions (Wzgarda-Raj *et al.* 2021).

48 Following our investigation on the reactivity of polypyridyl substrates towards ICl (Aragoni *et al.* 2008), we report here
49 on the structural and spectroscopic characterization of the novel salt 4,4'-disulfanediyldipyridinium chloride triiodide (**1**).

50 2. Structural commentary

51 By reacting 4,4'-dipyridyldisulfide (**L'**) and ICl in 1:1 molar ration, product **1** was isolated and characterized by
52 elemental analysis, melting point determination, FT—IR, and FT-Raman spectroscopy. Single crystal X-ray diffraction
53 analysis established **1** as (H₂L'²⁺)(Cl⁻)(I₃⁻) (Figure 1).

54 Compound **1** crystallizes in the monoclinic space group *P*2₁/*c* with four (H₂L'²⁺)(Cl⁻)(I₃⁻) units in the unit cell.

55 The asymmetric unit of compound **1** consists of a donor molecule protonated at both the N1 and N2 pyridine nitrogen
56 atoms H₂L'²⁺ counterbalanced by a chloride and a triiodide I₃⁻ anions. In the H₂L'²⁺ cation, the two pyridine rings are al-
57 most perpendicular [C1—S1—S2—C6 dihedral = 89.4 (1)°], being rotated by 2.7 (3) and 19.8 (3)° with respect to the re-
58 spective C—S—S plane. The linear triiodide anion [I1—I2—I3 = 177.13 (1)°] is remarkably asymmetric with a very short I1—
59 I2 distance [I1—I2 = 2.8179 (4)], close to the I—I distance of solid-state iodine (2.715 Å) (van Bolhuis *et al.* 1967), and a
60 longer one [I2—I3 = 3.0459 (4) Å].

61 3. Supramolecular features

62 The N—H terminal groups of the H₂L'²⁺ cation are involved in hydrogen bonding (HB) interactions with the chloride
63 anions [interaction a in Figure 1; a and c in Figure 2 and Table 1], thus forming a wavy 1-D H-bonded polymeric
64 structure that develops perpendicular to the *b*-axis. In addition, each chloride interacts with a terminal iodine atom of a
65 triiodide [I1⋯Cl = 3.4763 (8) Å; interaction b in Figures 1 and 2 and Table 1] at a distance shorter than the sum of the
66 relevant van der Waals radii (3.73 Å), so that the chloride and the triiodide could be considered to form a [I⋯I—I⋯Cl]²⁻
67 dianionic ensemble, unprecedented among the relevant polyinterhalides (Sonnenberg *et al.* 2020) deposited at the
68 Cambridge Structural Database. Nevertheless, the Cl⋯I distance is longer than those previously reported for the parent
69 [I₂Cl]⁻ anion (for example I—Cl = 3.158, 3.047 Å in the structures with CSD codes BEQXEA (Wang *et al.* 1999) and
70 BOJYIL (Pan *et al.* 2019), respectively) and [Cl₂I₂]²⁻ dianions (3.070 and 3.242 Å in DOXDOL (Buist & Kennedy, 2014)
71 and JUPCAA (Pan *et al.* 2015), respectively). These Cl⋯I interactions, shown in Figure 2, which fall into the realm of
72 halogen bonding (XB) interactions, generate the crystal packing along with a set of weak C—H⋯I contacts (entries d–g in
73 Table 1).

74 4. Conclusions

75 4,4'-Disulfanediyldipyridinium chloride triiodide (H₂L'²⁺)(Cl⁻)(I₃⁻)(**1**) was synthesized and characterized structurally and
76 spectroscopically. The isolation of **1** confirms that **L'** is not susceptible to the oxidative cleavage of the S—S disulfide
77 bond by diiodine and iodine monochloride under mild conditions, but that it can undergo protonation and template
78 fascinating supramolecular structures, as previously observed in the case of [(HL⁺)(I⁻)·5/2I₂]_∞. Further studies are
79 ongoing in our laboratory to investigate the reactivity of different dipyridyldichalcogenides towards dihalogens and

80 interhalogens and their versatility as building blocks for extended supramolecular assemblies based on σ -hole
81 interactions.

82 5. Synthesis and crystallization

83 All the reagents and solvents were used without further purification. Elemental analysis determinations were performed
84 with an EA1108 CHNS-O Fisons instrument. Fourier-Transform Infrared (FT—IR) spectroscopic measurements were
85 recorded on a Bruker IFS55 spectrometer at room temperature using a flow of dried air. Far-infrared (FIR; 500–50 cm^{-1})
86 spectra were recorded on polythene pellets using a Mylar beam-splitter and polythene windows (resolution 2 cm^{-1}).
87 Middle-infrared (MIR) spectra were recorded on KBr pellets, with a KBr beam-splitter and KBr windows (resolution 2
88 cm^{-1}). FT-Raman spectroscopy measurements were recorded on a Bruker RFS100 spectrometer (resolution of 2 cm^{-1}),
89 with an In–Ga–As detector operating with a Nd:YAG laser ($\lambda = 1064 \text{ nm}$) with a 180° scattering geometry (excitation
90 power 5 mW). Melting point determinations were carried out on a FALC mod. C apparatus.

91 To 2 ml of a CH_2Cl_2 solution of 4,4'-dipyridyldisulfide (19 mg, $8.6 \cdot 10^{-5}$ mol), a $0.054 \text{ mol} \cdot \text{L}^{-1}$ solution of ICl in the same
92 solvent was added dropwise in donor/ICl in 1:1 molar ratio. A brown crystalline precipitate was isolated from the mother
93 liquor by air-evaporation and washed with light petroleum ether. A small number of crystals were placed on a glass slide
94 and coated with a perfluoroether oil. A crystal suitable for X-ray diffraction analysis was selected and mounted on a glass
95 fiber. Elemental analysis calcd. for $\text{C}_{10}\text{H}_{10}\text{N}_2\text{S}_2\text{I}_3\text{Cl}$: 18.81; H, 1.57; N, 4.38; S, 10.04%. Found: C, 18.63; H, 1.78; N,
96 4.09, S 9.98%. *M.p.* > 240 $^\circ\text{C}$. FT-MIR (KBr pellet, 4000–400 cm^{-1}): 3854 s, 3460 s, 3437 s, 3088 s, 2743 s, 2363 s,
97 1952 s, 1846 s, 1773m, 1653 s, 1603 s, 1589 s, 1558m, 1441 s, 1371 s, 1277 s, 1086m, 1034m, 997m, 951m, 783m, 773 s,
98 617 s, 498m cm^{-1} . FT-FIR (polytene pellet, 500–50 cm^{-1}): 484m, 477m, 449w, 418m, 390w, 378m, 352m, 294w, 256m,
99 227 s, 170 s, 131m, 94m, 67m cm^{-1} . FT-Raman (500–50 cm^{-1} , 5 mW, relative intensities in parentheses related to the
100 highest peak taken equal to 10.0): 267(0.7), 155 (2.2), 137 (3.0), 113 (10.0) cm^{-1} .

101 6. Refinement

102 Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms bonded to heteroatoms
103 could be located from the difference Fourier map and their positions were freely refined. Other H atoms were placed in
104 geometrically calculated positions and were constrained to ride on their parent atom with $\text{C—H} = 0.95 \text{ \AA}$ and with
105 $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

106 **Table 1**

107 Intermolecular interactions (\AA , $^\circ$) of compound 1.

108 Interaction		A–B	B \cdots C	A \cdots C	A–B \cdots C
109 a	N1–H1 \cdots C11	0.82 (3)	2.41 (3)	3.101 (2)	142 (2)
110 b	I2–I1 \cdots C11	2.8179 (4)	3.4764 (8)	-	173.93 (2)
111 c	N2i–H2i \cdots C11	0.81 (4)	2.21 (4)	3.006 (3)	168
112 d	C10–H10 \cdots I3ii	0.95	3.07	3.833 (3)	138
113 e	C9–H9 \cdots I2ii	0.95	3.18	4.108 (4)	166
114 f	C7–H7 \cdots I2iii	0.95	3.03	3.738 (4)	132
115 g	C7–H7 \cdots I3iii	0.95	3.14	3.801 (3)	129

116 Symmetry codes: (i) $-1 + x, 3/2 - y, -1/2 + z$; (ii) $2 - x, 2 - y, 1 - z$; (iii) $+x, 3/2 - y, 1/2 + z$.

117 **Table 2**

118 Experimental details

119	Crystal data	
120	Chemical formula	$C_{10}H_{10}N_2S_2I_3Cl$
121	M_r	638.47
122	Crystal system, space group	Monoclinic, $P2_1/c$
123	Temperature (K)	120
124	a, b, c (Å)	12.9631 (11), 11.3802 (5), 13.1675 (11)
125	β (°)	117.624 (6)
126	V (Å ³)	1721.1 (2)
127	Z	4
128	Radiation type	Mo $K\alpha$
129	μ (mm ⁻¹)	5.83
130	Crystal size (mm)	0.32 × 0.22 × 0.06
131		
132	Data collection	
133	Diffractometer	Bruker-Nonius 95mm CCD camera on κ -goniostat
134	Absorption correction	Multi-scan <i>SADABS</i> V2.10 (Sheldrick, G.M., 2003)
135	T_{\min}, T_{\max}	0.632, 1.000
136	No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16769, 3959, 3621
137	R_{int}	0.027
138	$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.651
139		
140	Refinement	
141	$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.022, 0.046, 1.13
142	No. of reflections	3959
143	No. of parameters	170
144	H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
145	$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.74, -0.80

146 Computer programs: *DENZO* (Otwinowski & Minor, 1997) & *COLLECT* (Hooft, R.W.W., 1998), *SHELXT* 2018/2 (Sheldrick, 2018), *SHELXL* 2018/3
 147 (Sheldrick, 2015), *Olex2* 1.5 (Dolomanov *et al.*, 2009).

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 154 University of Southampton.

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206 *Chem.* 134, e202117587.
- 207 **Figure 1**
- 208 Ellipsoid plot of compound **1** with the numbering scheme adopted. Displacement ellipsoids were drawn at 50%
209 probability level. Labelled interactions are described according to Table 1.
- 210 **Figure 2**
- 211 Section of the crystal packing of compound **1** seen along the *c*-axis. Labelled contacts are described in Table 1.

1 supporting information

2 **Crystal structure of 4,4'-disulfanediylidipyridinium chloride triiodide**3 **Computing details**

4 Data collection: *COLLECT* (Hooft, R.W.W., 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) & *COLLECT*
 5 (Hooft, R.W.W., 1998); data reduction: *DENZO* (Otwinowski & Minor, 1997) & *COLLECT* (Hooft, R.W.W., 1998);
 6 program(s) used to solve structure: *SHELXT* 2018/2 (Sheldrick, 2018); program(s) used to refine structure: *SHELXL*
 7 2018/3 (Sheldrick, 2015); molecular graphics: Olex2 1.5 (Dolomanov *et al.*, 2009); software used to prepare material for
 8 publication: Olex2 1.5 (Dolomanov *et al.*, 2009).

9 **(I)**10 *Crystal data*

11	$C_{10}H_{10}N_2S_2I_3 \cdot Cl$	$F(000) = 1168$
12	$M_r = 638.47$	$D_x = 2.464 \text{ Mg m}^{-3}$
13	Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
14	$a = 12.9631 (11) \text{ \AA}$	Cell parameters from 17448 reflections
15	$b = 11.3802 (5) \text{ \AA}$	$\theta = 2.9\text{--}27.5^\circ$
16	$c = 13.1675 (11) \text{ \AA}$	$\mu = 5.83 \text{ mm}^{-1}$
17	$\beta = 117.624 (6)^\circ$	$T = 120 \text{ K}$
18	$V = 1721.1 (2) \text{ \AA}^3$	Cut-plate, brown
19	$Z = 4$	$0.32 \times 0.22 \times 0.06 \text{ mm}$

20 *Data collection*

21	Bruker-Nonius 95mm CCD camera on κ -goniostat diffractometer	3959 independent reflections 3621 reflections with $I > 2\sigma(I)$
22	Detector resolution: 9.091 pixels mm^{-1}	$R_{\text{int}} = 0.027$
23	φ & ω scans	$\theta_{\text{max}} = 27.6^\circ$, $\theta_{\text{min}} = 3.1^\circ$
24	Absorption correction: multi-scan <i>SADABS</i> V2.10 (Sheldrick, G.M., 2003)	$h = -16 \rightarrow 16$ $k = -14 \rightarrow 14$ $l = -16 \rightarrow 17$
25	$T_{\text{min}} = 0.632$, $T_{\text{max}} = 1.000$	
26	16769 measured reflections	

27 *Refinement*

28	Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
29	Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0127P)^2 + 2.0409P]$
30	$R[F^2 > 2\sigma(F^2)] = 0.022$	where $P = (F_o^2 + 2F_c^2)/3$
31	$wR(F^2) = 0.046$	$(\Delta/\sigma)_{\text{max}} = 0.003$
32	$S = 1.13$	$\Delta\rho_{\text{max}} = 0.74 \text{ e \AA}^{-3}$
33	3959 reflections	$\Delta\rho_{\text{min}} = -0.80 \text{ e \AA}^{-3}$
34	170 parameters	Extinction correction: <i>SHELXL2018/3</i> (Sheldrick 2018), $F_c^* = kFc[1 + 0.001x \text{Fc}^2\lambda^3/\sin(2\theta)]^{-1/4}$
35	0 restraints	Extinction coefficient: 0.00091 (7)
36	Primary atom site location: dual	
37	Hydrogen site location: mixed	

38 *Special details*

39 *Geometry.* All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

40 *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	
42	I1	0.67827 (2)	1.09634 (2)	0.41894 (2)	0.01952 (7)
43	I2	0.91260 (2)	1.16703 (2)	0.50892 (2)	0.01704 (6)
44	I3	1.16454 (2)	1.24311 (2)	0.59403 (2)	0.01907 (7)
45	Cl1	0.39992 (6)	0.98349 (6)	0.32707 (6)	0.01852 (15)
46	S2	0.78491 (6)	0.37523 (6)	0.66898 (6)	0.01882 (16)
47	S1	0.68199 (6)	0.41735 (6)	0.50222 (6)	0.01885 (16)
48	N2	1.1441 (2)	0.4996 (2)	0.7561 (2)	0.0192 (5)
49	H2	1.211 (3)	0.515 (3)	0.773 (3)	0.023*
50	N1	0.5417 (2)	0.7795 (2)	0.4871 (2)	0.0199 (5)
51	H1	0.516 (3)	0.847 (3)	0.477 (3)	0.024*
52	C6	0.9235 (2)	0.4272 (2)	0.6967 (2)	0.0158 (6)
53	C7	1.0175 (3)	0.3800 (3)	0.7912 (2)	0.0177 (6)
54	H7	1.005658	0.321243	0.836033	0.021*
55	C8	1.1276 (3)	0.4183 (3)	0.8198 (2)	0.0184 (6)
56	H8	1.192339	0.386896	0.885121	0.022*
57	C10	0.9433 (3)	0.5117 (3)	0.6312 (3)	0.0218 (6)
58	H10	0.880332	0.545123	0.565665	0.026*
59	C9	1.0561 (3)	0.5457 (3)	0.6637 (3)	0.0221 (6)
60	H9	1.071345	0.602813	0.619735	0.027*
61	C1	0.6298 (2)	0.5595 (2)	0.5052 (2)	0.0152 (6)
62	C5	0.5599 (3)	0.6079 (3)	0.3976 (3)	0.0198 (6)
63	H5	0.542253	0.564513	0.329894	0.024*
64	C2	0.6521 (2)	0.6235 (3)	0.6030 (2)	0.0180 (6)
65	H2A	0.698461	0.591158	0.676964	0.022*
66	C3	0.6063 (3)	0.7340 (3)	0.5911 (3)	0.0223 (7)
67	H3	0.620692	0.778684	0.657258	0.027*
68	C4	0.5170 (3)	0.7196 (3)	0.3912 (3)	0.0209 (6)
69	H4	0.469740	0.754210	0.318565	0.025*

70 *Atomic displacement parameters (\AA^2)*

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}	
72	I1	0.01655 (11)	0.02240 (11)	0.02038 (11)	0.00155 (7)	0.00921 (9)	0.00205 (8)
73	I2	0.01911 (11)	0.01738 (10)	0.01659 (11)	0.00135 (7)	0.00995 (8)	0.00154 (7)
74	I3	0.01743 (11)	0.02150 (11)	0.01500 (11)	-0.00096 (7)	0.00473 (8)	-0.00102 (7)
75	Cl1	0.0157 (3)	0.0192 (3)	0.0216 (4)	0.0040 (3)	0.0095 (3)	0.0045 (3)
76	S2	0.0134 (4)	0.0209 (3)	0.0225 (4)	0.0021 (3)	0.0086 (3)	0.0052 (3)
77	S1	0.0150 (4)	0.0173 (3)	0.0196 (4)	0.0037 (3)	0.0042 (3)	-0.0033 (3)
78	N2	0.0117 (12)	0.0193 (12)	0.0274 (14)	-0.0021 (10)	0.0098 (11)	-0.0063 (11)
79	N1	0.0167 (13)	0.0164 (12)	0.0264 (14)	0.0037 (10)	0.0099 (11)	0.0005 (11)
80	C6	0.0150 (14)	0.0162 (13)	0.0173 (14)	0.0015 (11)	0.0083 (12)	-0.0029 (11)
81	C7	0.0193 (15)	0.0203 (14)	0.0148 (14)	0.0041 (12)	0.0091 (12)	0.0013 (12)
82	C8	0.0148 (15)	0.0221 (14)	0.0165 (14)	0.0033 (11)	0.0056 (12)	-0.0029 (12)
83	C10	0.0186 (16)	0.0207 (15)	0.0225 (15)	0.0042 (12)	0.0064 (13)	0.0084 (12)
84	C9	0.0205 (16)	0.0199 (14)	0.0282 (17)	-0.0015 (12)	0.0131 (14)	0.0028 (13)
85	C1	0.0084 (13)	0.0163 (13)	0.0187 (14)	-0.0010 (10)	0.0043 (11)	-0.0023 (11)
86	C5	0.0175 (15)	0.0221 (15)	0.0177 (15)	0.0029 (12)	0.0064 (12)	-0.0027 (12)
87	C2	0.0153 (15)	0.0191 (14)	0.0147 (14)	0.0007 (11)	0.0027 (12)	0.0000 (12)
88	C3	0.0200 (16)	0.0199 (14)	0.0229 (16)	-0.0006 (12)	0.0065 (13)	-0.0079 (13)

89	C4	0.0194 (16)	0.0228 (15)	0.0207 (15)	0.0036 (12)	0.0094 (13)	0.0043 (13)
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90 *Geometric parameters (Å, °)*

91	I1—I2	2.8180 (4)	C7—C8	1.368 (4)
92	I2—I3	3.0459 (4)	C8—H8	0.9500
93	S2—S1	2.0285 (11)	C10—H10	0.9500
94	S2—C6	1.762 (3)	C10—C9	1.375 (4)
95	S1—C1	1.762 (3)	C9—H9	0.9500
96	N2—H2	0.81 (3)	C1—C5	1.394 (4)
97	N2—C8	1.331 (4)	C1—C2	1.387 (4)
98	N2—C9	1.330 (4)	C5—H5	0.9500
99	N1—H1	0.82 (3)	C5—C4	1.374 (4)
100	N1—C3	1.334 (4)	C2—H2A	0.9500
101	N1—C4	1.337 (4)	C2—C3	1.368 (4)
102	C6—C7	1.384 (4)	C3—H3	0.9500
103	C6—C10	1.393 (4)	C4—H4	0.9500
104	C7—H7	0.9500		
105				
106	I1—I2—I3	177.129 (8)	C9—C10—H10	120.8
107	C6—S2—S1	103.93 (10)	N2—C9—C10	120.7 (3)
108	C1—S1—S2	105.03 (10)	N2—C9—H9	119.6
109	C8—N2—H2	116 (2)	C10—C9—H9	119.6
110	C9—N2—H2	122 (2)	C5—C1—S1	114.5 (2)
111	C9—N2—C8	122.0 (3)	C2—C1—S1	125.9 (2)
112	C3—N1—H1	123 (2)	C2—C1—C5	119.6 (3)
113	C3—N1—C4	122.1 (3)	C1—C5—H5	120.6
114	C4—N1—H1	115 (2)	C4—C5—C1	118.8 (3)
115	C7—C6—S2	116.4 (2)	C4—C5—H5	120.6
116	C7—C6—C10	119.1 (3)	C1—C2—H2A	120.6
117	C10—C6—S2	124.5 (2)	C3—C2—C1	118.9 (3)
118	C6—C7—H7	120.2	C3—C2—H2A	120.6
119	C8—C7—C6	119.6 (3)	N1—C3—C2	120.5 (3)
120	C8—C7—H7	120.2	N1—C3—H3	119.8
121	N2—C8—C7	120.0 (3)	C2—C3—H3	119.8
122	N2—C8—H8	120.0	N1—C4—C5	120.1 (3)
123	C7—C8—H8	120.0	N1—C4—H4	120.0
124	C6—C10—H10	120.8	C5—C4—H4	120.0
125	C9—C10—C6	118.4 (3)		
126				
127	S2—S1—C1—C5	177.6 (2)	C7—C6—C10—C9	-0.4 (4)
128	S2—S1—C1—C2	-2.7 (3)	C8—N2—C9—C10	0.9 (4)
129	S2—C6—C7—C8	-178.3 (2)	C10—C6—C7—C8	1.1 (4)
130	S2—C6—C10—C9	178.9 (2)	C9—N2—C8—C7	-0.2 (4)
131	S1—S2—C6—C7	-160.8 (2)	C1—C5—C4—N1	-0.5 (4)
132	S1—S2—C6—C10	19.9 (3)	C1—C2—C3—N1	-0.2 (4)
133	S1—C1—C5—C4	-178.7 (2)	C5—C1—C2—C3	-1.2 (4)
134	S1—C1—C2—C3	179.1 (2)	C2—C1—C5—C4	1.5 (4)
135	C6—C7—C8—N2	-0.8 (4)	C3—N1—C4—C5	-0.9 (5)
136	C6—C10—C9—N2	-0.5 (4)	C4—N1—C3—C2	1.3 (5)
